GLYOXAL CROSSLINKED CELLULOSIC FIBERS HAVING IMPROVED BRIGHTNESS AND COLOR

FIELD OF THE INVENTION

The present invention relates generally to cellulosic fibers and, more specifically, to glyoxal crosslinked cellulosic fibers having improved brightness and color.

BACKGROUND OF THE INVENTION

Cellulosic fibers are a basic component of absorbent products such as diapers. The ability of an absorbent product containing cellulosic fibers to initially acquire and distribute liquid will generally depend on the product's dry bulk and capillary structure. However, the ability of a product to acquire additional liquid on subsequent insults will depend on the product's wet bulk. Cellulosic fibers, although absorbent, tend to collapse on wetting and to retain absorbed liquid near the point of liquid insult. The inability of wetted cellulosic fibers in absorbent products to further acquire and distribute liquid to sites remote from liquid insult can be attributed to a diminished acquisition rate due in part to the loss of fiber bulk associated with liquid absorption. Absorbent products made from cellulosic fluff pulp, a form of cellulosic fibers having an extremely high void volume, lose bulk on liquid acquisition and the ability to further wick and acquire liquid, causing local saturation.

Crosslinked cellulosic fibers generally have enhanced wet bulk compared to uncrosslinked fibers. The enhanced bulk is a consequence of the stiffness, twist, and curl imparted to the fiber as a result of crosslinking. Accordingly, crosslinked fibers are

advantageously incorporated into absorbent products to enhance their wet bulk and liquid acquisition rate and to also reduce rewet.

Some of the first crosslinked cellulosic fibers were prepared by treating cellulosic fibers with formaldehyde and various formaldehyde addition products. See, for example, U.S. Patent No. 3,224,926; U.S. Patent No. 3,241,553; U.S. Patent No. 3,932,209; U.S. Patent No. 4,035,147; and U.S. Patent No. 3,756,913. Unfortunately, the irritating effect of formaldehyde vapor on the eyes and skin is a marked disadvantage of the fibers. In addition, such crosslinked fibers typically exhibit objectionable odor and have low fiber brightness.

Alternatives to formaldehyde and formaldehyde addition product crosslinking agents have been developed. Among these are dialdehyde crosslinking agents. See, for example, U.S. Patent No. 4,822,453, which describes absorbent structures containing individualized, crosslinked fibers, wherein the crosslinking agent is selected from the group consisting of C_2-C_9 dialdehydes, with glutaraldehyde being preferred. The reference appears to overcome many of the disadvantages associated with formaldehyde and/or formaldehyde addition products. However, the cost associated with producing fibers crosslinked with dialdehyde crosslinking agents such as glutaraldehyde is considered too high to result in significant commercial success. Therefore, further efforts have been made to find cellulosic fiber crosslinking agents that are both safe for use on the human skin, have good aesthetics (low odor and high fiber brightness), and are commercially feasible.

Polycarboxylic acids have been used to crosslink cellulosic fibers. See, for example, U.S. Patent No. 5,137,537; U.S. Patent No. 5,183,707; and U.S. Patent No. 5,190,563. These references describe absorbent structures containing individualized cellulosic fibers crosslinked with a C_2-C_9 polycarboxylic acid. The ester crosslink bonds formed by the polycarboxylic acid crosslinking agents differ from the acetal crosslink bonds that result from the mono- and di-aldehyde crosslinking agents. Absorbent structures made from these individualized, ester-crosslinked fibers exhibit increased dry and wet resilience and have improved responsiveness to wetting relative to structures containing uncrosslinked fibers. Furthermore, the preferred polycarboxylic crosslinking agent, citric acid, is available in large quantities at relatively low prices making it commercially competitive with formaldehyde

and formaldehyde addition products. Unfortunately, the preferred C_2-C_9 crosslinking agent, citric acid, can cause discoloring (i.e., yellowing) of the white cellulosic fibers when treated at elevated temperatures required for crosslinking. In addition, unpleasant odors can also be associated with the use of α-hydroxy carboxylic acids such as citric acid. The above-noted references do not describe processes that reduce the odor or increase the brightness of the treated fibers.

More recently, it was found that the characteristic odor associated with citric acid crosslinked cellulosic fibers could be removed and the brightness improved by contacting the fibers with an alkaline solution (e.g., an aqueous solution of sodium hydroxide) and an oxidizing bleaching agent (e.g., hydrogen peroxide). See U.S. Patent No. 5,562,740. In the method, the alkaline solution raises the finished fiber pH preferably to the 5.5-6.5 range from about 4.5. This in combination with the oxidizing bleaching agent eliminates the "smokey and burnt" odor characteristics of the crosslinked fibers. The oxidizing bleaching agent when added at high consistency increases the final product brightness to 80 to 86 from 70 to 75 and reduces odor.

Although some disadvantages related to brightness and color associated with crosslinked cellulosic fibers have been addressed, a need remains for cellulosic fibers having the advantages of bulk, liquid acquisition, and rewet associated with crosslinked cellulosic fibers without the disadvantages related to diminished fiber brightness and color. The present invention seeks to fulfill these needs and provides further related advantages.

SUMMARY OF THE INVENTION

In one aspect, the present invention provides individualized cellulosic fibers having improved brightness and color. The cellulosic fibers of the invention are intrafiber crosslinked cellulosic fibers obtainable from cellulosic fibers by treatment with glyoxal. The cellulosic fibers of the invention are crosslinked by treatment with only an aqueous glyoxal solution (e.g., without a crosslinking catalyst). The fibers of the present invention have a brightness greater than about 80% ISO and color characterized by an L value greater than about 92 and a b value less than about 7.5.

In another aspect of the invention, methods for the preparation of cellulosic fibers having improved brightness and color are provided. In the methods, a fibrous web of

cellulosic fibers is treated with only an aqueous glyoxal solution (e.g., without a crosslinking catalyst), wet fiberized, and then dried and cured to provide individualized cellulosic fibers having improved brightness and color.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

The present invention provides cellulosic fibers having improved brightness and color and methods for their preparation. The fibers of the invention are glyoxal crosslinked cellulosic fibers. The glyoxal crosslinked cellulosic fibers are intrafiber crosslinked fibers. The glyoxal crosslinked cellulosic fibers are made by treatment with an amount of glyoxal effective to provide crosslinked fibers having a brightness greater than about 80% ISO. The glyoxal crosslinked cellulosic fibers have a color characterized by an L value greater than about 92, an a value greater than about -2.3 and less than about +0.2, and a b value less than about 7.5.

As used herein, the term "glyoxal crosslinked cellulosic fibers" refers to cellulosic fibers obtainable from cellulose fibers by treatment with an aqueous glyoxal solution without the use of a crosslinking catalyst. The glyoxal crosslinked cellulosic fibers of the invention are made by treating a mat or web of cellulosic fibers with an aqueous glyoxal solution to provide glyoxal treated fibers, which are then separated into individual glyoxal treated fibers, and heated for a time and at a temperature to effect curing (i.e., to provide glyoxal crosslinked cellulosic fibers). The glyoxal crosslinked fibers of the invention are made by treatment with an aqueous glyoxal solution without the use of a crosslinking catalyst. Representative methods for making the glyoxal crosslinked cellulosic fibers of the invention are described in Example 1.

As used herein, the term "brightness" refers to the reflectance of blue light corresponding to a centroid wavelength of 457 nm in terms of the perfect reflecting diffuser (perfect reflecting diffuser is the ideal reflecting surface that neither absorbs nor transmits light, but reflects diffusely, with the radiance of the reflecting surface being the same for all reflecting angles, regardless of the angular distribution of the incident light). Brightness was measured according to TAPPI T 525 om-02 on a Technibrite MicroTB-1C instrument (Technydine Corp.).

In one embodiment, the glyoxal crosslinked fibers have a brightness greater than about 75% ISO. In another embodiment, the glyoxal crosslinked fibers have a brightness greater than about 80% ISO. In another embodiment, the glyoxal crosslinked fibers have a brightness greater than about 85% ISO. The brightness of representative glyoxal crosslinked cellulosic fibers of the invention as a function of crosslinking time and temperature is presented in Tables 1 and 2.

In addition to high brightness, the glyoxal crosslinked fibers of the invention advantageous exhibit improved color properties as indicated by the Opponent colors scales (*L*, *a*, *b)*, and Whiteness index (WI_{CDM-L}) values. *L*, *a* and *b* are used to designate measured values of three attributes of surface-color appearance as follows: *L* represents lightness, increasing from zero for black to 100 for perfect white; *a* represents redness when positive, greenness when negative, and zero for gray; and *b* represents yellowness when positive, blueness when negative, and zero for gray. The concept of opponent colors was proposed by Hering in 1878. Starting in 1940s, a number of measurable *L, a, b* dimensions have been defined by equations relating them to the basic CIE XYZ tristimulus quantities defined in CIE Document No. 15. Measured values for a given color will depend on color space in which they are expressed [(TAPPI T 1213 sp-98 "Optical measurements terminology (related to appearance evaluation of paper")].

In one embodiment, the glyoxal crosslinked fibers have a color characterized by an *L* value greater than about 92, an *a* value greater than about -2.3 and less than about +0.2, and a *b* value less than about 7.5, and a Whiteness Index from about 72 to about 86. In one embodiment, the glyoxal crosslinked fibers have a color characterized by an *L* value greater than about 95. The color properties of representative glyoxal crosslinked cellulosic of the invention are provided in Tables 1 and 2. Whiteness Index (WI) is determined using a color difference meter (CDM) and is defined as:

$$WI_{[CDM-L]} = L\text{-}3b.$$

Basic color measurement is made using commercially available instruments (e.g., Technibrite MicroTB-1C, Technydine Corp.). The instrument scans through the brightness and color filters. Fifty readings are taken at each filter position and averaged and the resulting values are printed out as Brightness, R(X), R(Y), and R(Z). Brightness is ISO

brightness (457 nm), R(X) is absolute red reflectance (595 nm), R(Y) is absolute green reflectance (557 nm), and R(Z) is absolute blue reflectance (455 nm). The CIE tristimulus functions X, Y, and Z are then computed in accordance with the following equations: X = 0.782 R(X) + 0.198 R(Z); Y = R(Y); and Z = 1.181 R(Z). Next *L*, *a* and *b* values are computed using the established equations (Technibrite Micro TB-1C Instruction Manual TTM 575-08, Oct. 30, 1989). $WI_{(CDM-L)}$ was calculated according to the equation: $WI_{(CDM-L)} = L - 3b$, according to TAPPI T 1216 sp-98 (TAPPI T 1216 sp-98 "Indices for whiteness, yellowness, brightness and luminous reflectance factor").

To further illustrate the principles of the invention, a discussion of whiteness and brightness is useful. Webster's Dictionary defines white as "the object color of greatest lightness characteristically perceived to belong to objects that reflect diffusely nearly all incident energy throughout the visible spectrum". Used as a noun or adjective, white is defined as "free from color". Most natural and many man-made products are never "free from color". Whether the "white" product is fluff pulp, paper, textiles, plastics, or teeth, there is usually an intrinsic color, other than white, associated with it. Consider two hypothetical objects, the first that meets Webster's definition of white: one characterized by a flat spectrum of high reflectance and a second, which is the first with a small amount of blue colorant added (results in an unequal spectrum). Most people will judge the second as being the whiter of the two even though its total reflectance is lower in certain spectral regions. The first will be judged as a "yellow-white" while the second a "blue-white". Human color vision is more than just a sensation. It is also quite subjective and certain associations are unconsciously made. Blue-white is associated with "clean and pure", while "yellow-white" denotes "dirty, old or impure". The type and amounts of fillers and colorants to use, which hues are appropriate (e.g., red-blue, green-blue), and the optimal optical prescription to target have been the subject of considerable interest.

The preparation of cellulosic fibers crosslinked with glyoxal using a crosslinking catalyst is described in Example 2. The crosslinking procedure described in Example 2 is as described in Example 1 of U.S. Patent No. 4,888,093, Individualized Crosslinked-Fibers and Process for Making Said Fibers. The crosslinked cellulosic fibers prepared as described in Example 2 had a brightness significantly lower than the brightness of the glyoxal crosslinked

fibers of the invention. In contrast to the glyoxal crosslinked cellulosic fibers of the invention having a brightness greater than about 80% ISO, the highest brightness achieved for the fibers crosslinked with glyoxal and crosslinking catalyst was about 74% ISO.

In another aspect, the present invention provides a method for making cellulosic fibers crosslinked by treatment with an aqueous glyoxal solution without the use of a crosslinking catalyst. In the method, cellulosic fibers are treated with an effective amount of glyoxal to achieve the brightness and color enhancements described herein. Generally, the fibers are treated with from about 0.5 to about 5 percent by weight glyoxal based on the total weight of the treated fibers. In one embodiment, the fibers are treated with from about 1 to about 4 percent by weight based on the weight of fibers.

In general, the cellulose fibers of the present invention may be prepared by a system and apparatus as described in U.S. Patent No. 5,447,977 to Young, Sr. et al., which is incorporated herein by reference in its entirety. Briefly, the fibers are prepared by a system and apparatus that includes a conveying device for transporting a mat or web of cellulose fibers through a fiber treatment zone; an applicator for applying a treatment substance such as an aqueous glyoxal solution from a source to the fibers at the fiber treatment zone; a fiberizer for separating the individual cellulose fibers comprising the mat to form a fiber output comprised of substantially unbroken and essentially singulated cellulose fibers; a dryer coupled to the fiberizer for flash evaporating residual moisture; and a controlled temperature zone for additional heating of fibers and an oven for curing the crosslinking agent, to form dried and cured individualized crosslinked fibers.

As used herein, the term "mat" refers to any nonwoven sheet structure comprising cellulose fibers or other fibers that are not covalently bound together. The fibers include fibers obtained from wood pulp or other sources including cotton rag, hemp, grasses, cane, husks, cornstalks, or other suitable sources of cellulose fibers that may be laid into a sheet. The mat of cellulose fibers is preferably in an extended sheet form, and may be one of a number of baled sheets of discrete size or may be a continuous roll.

Each mat of cellulose fibers is transported by a conveying device, for example, a conveyor belt or a series of driven rollers. The conveying device carries the mats through the fiber treatment zone.

At the fiber treatment zone, an aqueous glyoxal solution is applied to the cellulose fibers. The crosslinking solution is preferably applied to one or both surfaces of the mat using any one of a variety of methods known in the art, including spraying, rolling, or dipping. Once the glyoxal solution has been applied to the mat, the crosslinking solution may be uniformly distributed through the mat, for example, by passing the mat through a pair of rollers.

After the fibers have been treated with the crosslinking agent, the impregnated mat is fiberized by feeding the mat through a hammermill. The hammermill serves to disintegrate the mat into its component individual cellulose fibers, which are then air conveyed through a drying unit to remove the residual moisture. In a preferred embodiment, the fibrous mat is wet fiberized.

The pulp is then air conveyed through an additional heating zone to bring the temperature of the pulp to the cure temperature. The cure temperature for glyoxal is about 150°C. In one embodiment, the dryer comprises a first drying zone for receiving the fibers and for removing residual moisture from the fibers via a flash-drying method, and a second heating zone for curing the crosslinking agent. Alternatively, in another embodiment, the treated fibers are blown through a flash-dryer to remove residual moisture, heated to a curing temperature, and then transferred to an oven where the treated fibers are subsequently cured. Overall, the treated fibers are dried and then cured for a sufficient time and at a sufficient temperature to effect crosslinking. Typically, the fibers are oven-dried and cured for about 1 to about 20 minutes at a temperature from about 120°C to about 165°C.

As noted above, the present invention relates to crosslinked cellulose fibers. Although available from other sources, cellulosic fibers useful for making glyoxal crosslinked cellulosic fibers of the invention are derived primarily from wood pulp. Suitable wood pulp fibers for use with the invention can be obtained from well-known chemical processes such as the kraft and sulfite processes, with or without subsequent bleaching. The pulp fibers may also be processed by thermomechanical, chemithermomechanical methods, or combinations thereof. The preferred pulp fiber is produced by chemical methods. Ground wood fibers, recycled or secondary wood pulp fibers, and bleached and unbleached wood pulp fibers can be used. The preferred starting material is prepared from long-fiber

coniferous wood species, such as southern pine, Douglas fir, spruce, and hemlock. Details of the production of wood pulp fibers are well-known to those skilled in the art. These fibers are commercially available from a number of companies, including Weyerhaeuser Company. For example, suitable cellulose fibers produced from southern pine that are usable with the present invention are available from Weyerhaeuser Company under the designations CF416, CF405, NF405, PL416, FR416, FR516, and NB416.

The wood pulp fibers useful in the present invention can also be pretreated prior to use with the present invention. This pretreatment may include physical treatment, such as subjecting the fibers to steam or chemical treatment. Although not to be construed as a limitation, examples of pretreating fibers include the application of fire retardants to the fibers, and surfactants or other liquids, such as solvents, which modify the surface chemistry of the fibers. Other pretreatments include incorporation of antimicrobials, pigments, and densification or softening agents. Fibers pretreated with other chemicals, such as thermoplastic and thermosetting resins also may be used. Combinations of pretreatments also may be employed.

Method for determining fiber brightness. The brightness (% ISO) of cellulosic fibers crosslinked with glyoxal of the invention was determined by TAPPI T 525 om-02.

The brightness, *L, a, b*, and WI values of representative glyoxal crosslinked fibers of the invention as a function of glyoxal addition, cure temperature, and cure time are summarized in Tables 1 and 2.

Table 1. Brightness, *L, a, b*, and WI for Representative Glyoxal Crosslinked Fibers.

Glyoxal (%)	Cure Temp. (°C)	Cure Time (min.)	Brightness (% ISO)	*L*	*a*	*b*	$WI_{(CDM-L)}$
1	150	5	84.2	95.3	-0.52	5.16	79.8
1	150	7	82.3	94.6	-0.57	5.71	77.5
2	130	3	87.4	95.9	-0.66	3.50	85.4
2	130	5	87.0	95.9	-0.70	3.78	84.5
2	130	7	86.0	95.7	-0.76	4.22	83.0
2	140	3	87.6	96.2	-0.71	3.75	84.9

Glyoxal (%)	Cure Temp. (°C)	Cure Time (min.)	Brightness (% ISO)	*L*	*a*	*b*	$WI_{(CDM-L)}$
2	140	5	85.1	95.6	-0.80	4.90	80.9
2	140	7	84.5	95.4	-0.72	5.00	83.0
2	150	3	86.0	95.8	-0.71	4.39	82.6
2	150	5	82.8	94.7	-0.75	5.44	78.4
2	150	5	83.2	95.1	-0.71	5.62	78.3
2	150	7	82.6	95.0	-0.69	5.90	77.3
2.5	130	3	87.2	95.9	-0.67	3.60	85.1
2.5	130	5	87.0	95.8	-0.73	3.76	84.6
2.5	130	7	86.8	96.0	-0.72	4.08	83.7
2.5	140	3	86.9	95.8	-0.75	3.76	84.5
2.5	140	5	86.7	96.1	-0.82	4.26	83.3
2.5	140	7	84.7	95.6	-0.75	5.09	83.7
2.5	150	3	86.9	95.9	-0.73	3.85	84.3
2.5	150	5	83.6	94.3	-0.76	4.97	80.0
2.5	150	7	80.7	94.9	-0.75	6.41	75.1
3	130	3	87.5	96.0	-0.71	3.62	85.2
3	130	5	87.8	96.1	-0.71	3.53	85.6
3	130	7	86.7	96.0	-0.77	4.25	83.3
3	140	3	87.4	96.0	-0.74	3.71	84.9
3	140	5	86.2	96.0	-0.81	4.55	82.3
3	140	7	86.2	95.8	-0.72	4.26	83.3
3	150	3	86.3	95.9	-0.77	4.45	82.6
3	150	5	83.6	95.2	-0.67	5.58	78.5
3	150	5	83.4	95.4	-0.81	5.90	77.7
3	150	7	80.1	94.6	-0.48	7.26	72.8
3	150	7	81.2	94.7	-0.82	6.58	75.0
3.5	130	3	87.7	96.0	-0.73	3.44	85.7
3.5	130	5	87.7	96.2	-0.76	3.77	84.9
3.5	130	7	86.8	96.0	-0.77	4.16	83.5
3.5	140	3	87.6	96.2	-0.75	3.86	84.7
3.5	140	5	85.8	95.7	-0.84	4.41	82.5
3.5	140	7	85.2	95.7	-0.82	4.96	83.5
3.5	150	3	85.7	95.8	-0.80	4.66	81.8
3.5	150	5	82.8	95.3	-0.80	6.15	76.8
3.5	150	7	81.9	94.9	-0.79	6.32	76.0
4	130	3	87.6	95.9	-0.69	3.37	85.8
4	130	5	88.0	96.3	-0.69	3.60	85.5
4	130	7	87.3	96.1	-0.76	3.95	84.2

Glyoxal (%)	Cure Temp. (°C)	Cure Time (min.)	Brightness (% ISO)	*L*	*a*	*b*	$WI_{(CDM-L)}$
4	140	3	88.0	96.3	-0.73	3.61	85.5
4	140	5	86.8	96.1	-0.82	4.23	83.4
4	140	7	85.3	95.8	-0.82	4.97	84.2
4	150	3	87.3	96.2	-0.77	3.97	84.2
4	150	5	83.3	94.8	-0.78	5.18	79.3
4	150	5	84.5	95.7	-0.89	5.36	79.6
4	150	7	83.4	95.3	-0.76	5.65	78.3
5	130	7	80.6	94.7	-0.69	7.10	73.4
5	150	5	83.8	95.5	-0.69	5.78	78.1

Table 2. Brightness, *L*, *a*, *b*, and WI Values for Representative Glyoxal Crosslinked Fibers.

Glyoxal (%)	Cure Temp. (°C)	Cure Time (min.)	Brightness (% ISO)	*L*	*a*	*b*	$WI_{(CDM-L)}$
2	141	3	87.3	96.2	-0.62	4.04	84.1
2	141	5	86.3	96.0	-0.62	4.54	82.4
2	141	7	84.5	95.5	-0.60	5.29	79.6
2	149	3	86.5	96.0	-0.64	4.41	82.8
2	149	5	84.0	95.4	-0.56	5.49	78.9
2	149	7	80.5	94.5	-0.36	6.82	74.0
2	157	3	83.7	95.3	-0.55	5.63	78.4
2	157	5	80.9	94.6	-0.39	6.71	74.5
2	157	7	74.3	92.6	0.01	8.99	65.6
3	141	3	87.2	96.1	-0.64	4.05	84.0
3	141	5	85.8	95.8	-0.65	4.68	81.8
3	141	7	84.0	95.4	-0.64	5.40	79.2
3	149	3	85.9	95.7	-0.69	4.43	82.4
3	149	5	84.3	95.4	-0.63	5.31	79.5
3	149	7	81.6	94.8	-0.51	6.38	75.7
3	157	3	83.0	95.2	-0.66	5.88	77.6
3	157	5	79.0	94.1	-0.47	7.45	71.8
3	157	7	72.8	92.1	-0.03	9.50	63.6

The present invention provides cellulosic fibers having improved brightness and color. The fibers of the invention are intrafiber crosslinked cellulosic fibers obtainable from cellulosic fibers by treatment with an aqueous glyoxal solution. The crosslinked fibers can be formed from cellulosic fibers by treatment with an amount of glyoxal without the use of a crosslinking catalyst effective to provide the brightness and color enhancement described herein.

The glyoxal crosslinked cellulosic fibers of the present invention can be advantageously incorporated into an absorbent product. Such products can further include other fibers such as fluff pulp fibers, synthetic fibers, other crosslinked fibers, and absorbent materials such as superabsorbent polymeric materials. Representative absorbent products

that can include the fibers of the invention include infant diapers, adult incontinence products, and feminine hygiene products. The fibers can be included in liquid acquisition, distribution, or storage layers. The glyoxal crosslinked cellulosic fibers of the present invention can be advantageously incorporated into tissue and towel products.

The fibers of the invention can be advantageously incorporated into paperboard products, including single and multi-ply paperboard products. Paperboard products that include the fibers can be used in insulation applications, for example, insulated cups and containers. Paperboard products that include the fibers can also be used as packaging materials.

The glyoxal crosslinked cellulosic fibers of the invention are essentially odorless. This is in contrast to cellulosic fibers that have been modified with polycarboxylic acid crosslinking agents such as citric acid crosslinked cellulosic fibers.

The following examples are for the purposes of illustrating, not limiting, the present invention.

EXAMPLES

Example 1

Representative Glyoxal Crosslinked Cellulosic Fibers

In this example, methods for forming representative high bulk fibers in accordance with the present invention are described.

Method A. A selected amount of glyoxal (CARTABOND GHF containing 40 percent by weight glyoxal in water) was applied to both sides of a twenty gram pulp sheet (CF416, dried wood pulp fibers available from Weyerhaeuser Co.) using a 5 mL disposable syringe and 23.1 gauge needle. The sample was held in a resealable plastic bag for 16-18 hours at room temperature, then broken into pieces (e.g., about 2x2 cm), passed through a laboratory fiberizer, and collected as a loose pad. The pad was broken into small pieces (e.g., about 3x3 cm), placed into a screen basket and cured at a fixed temperature and time in a Despatch V Series oven.

Glyoxal crosslinked fibers prepared by this method have the brightness, *L*, *a*, *b*, and WI values described in Table 1.

Method B. Pulp sheets in roll form (CF416, dried wood pulp fibers available from Weyerhaeuser Co.) were treated with glyoxal (commercially available under the designation CARTABOND GHF from Clariant Corp.) according to the following procedure. The pulp sheet was fed from the roll through a constantly replenished bath of the crosslinking solution (i.e., an aqueous solution containing a glyoxal concentration determined by the weight add-on desired), then through a roll nip set to remove sufficient solution so that the pulp sheet after treating was at about 40% moisture content. The concentration of the bath was adjusted to achieve the desired level of chemical addition to the pulp sheet. After the roll nip, the wet sheet was fed through a hammer mill to fiberize the pulp. The individualized fibers were then blown through a flash dryer to affect drying and then to a cyclone where the treated cellulose fluff was separated from the air stream. The pulp was air conveyed through an additional heating zone to bring the temperature of the pulp to the cure temperature and then transferred to an oven where the treated fibers were subsequently cured.

Glyoxal crosslinked fibers prepared by this method have the brightness, *L*, *a*, *b*, and WI values described in Table 2.

Example 2

Comparative Example: Brightness and Color for Cellulose Fibers Crosslinked with Glyoxal Using Catalyst

In this example, the brightness and color of cellulose fibers crosslinked with glyoxal and catalyst is described. Wood pulp fibers (never-dried FR416, available from Weyerhaeuser Co.) were treated with glyoxal and crosslinking catalyst, zinc nitrate hexahydrate, 30 weight percent based on the weight of glyoxal, according to the procedure described in U.S. Patent No. 4,888,093, Individualized Crosslinked-Fibers and Process for Making Said Fibers, Example 1. The only difference was that only one of the solutions was adjusted to 3.7 because all others were below this value.

The fibers were treated with glyoxal (3, 6, and 9 weight percent) and catalyst and then cured (145°C, 45 min) to provide glyoxal crosslinked fibers. The brightness (% ISO) and color (*L*, *a*, *b*, and WI) values for the glyoxal crosslinked fibers is summarized in Table 3.

Table 3. Brightness, *L*, *a*, *b*, and WI for Glyoxal/Catalyst Crosslinked Fibers

Glyoxal (%)	Cure Temp. (°C)	Cure Time (min.)	Brightness (% ISO)	*L*	*a*	*b*	$WI_{(CDM-L)}$
3	145	45	73.2	92.5	-0.59	9.54	63.9
6	145	45	65.6	90.3	-0.31	12.81	51.8
6*	145	45	67.4	90.3	-0.23	11.43	56.0
9	145	45	60.6	88.0	0.06	13.89	46.3

*pH was adjusted to 3.7 (as specified in the '093 patent). The pH of the other three samples remained 'as-is', 2.74, 2.50, and 2.27 for 3, 6, 9 wt% glyoxal, respectively.

As shown in Table 3, brightness and *L* decrease with increasing glyoxal amount, and *b* increases with increasing glyoxal amount.

While the preferred embodiment of the invention has been illustrated and described, it will be appreciated that various changes can be made therein without departing from the spirit and scope of the invention.